MEASUREMENT OF INFRARED SPECTRAL DIRECTIONAL HEMISPHERICAL REFLECTANCE AND EMISSIVITY AT BNM-LNE[1]

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ABSTRACT

An infrared reflectometer has been designed by BNM-LNE (Bureau National de Métrologie – Laboratoire National d'Essais) to measure the directional hemispherical spectral reflectance of solid materials at ambient temperature. For opaque materials, the directional spectral emissivity can be calculated from the measured reflectance. The reflectance can be measured from 0.8 µm to 14 µm in 5 directions with an angle of 12°, 24°, 36°, 48° and 60° with respect to the normal to the surface of the sample. The optical arrangement to collect the reflected flux is based on the Coblentz's arrangement (hemispherical mirror). In fact, four mirrors cut in an hemisphere are used to collect the flux reflected by the sample. This optical arrangement was chosen to limit the angle of incidence of rays on the detector (38° instead of 90° for the Coblentz's arrangement). The uncertainties have been evaluated. The final expanded uncertainty (level of confidence 95%) is estimated at about +/- 0.03 for wavelengths between 0.8 µm to 10 µm and +/- 0.04 for wavelengths over 10 µm. The values of the spectral reflectance measured on a black paint and on a white ceramic tile are compared to those measured by 2 other laboratories (PTB and NIST). The results validate the measurements performed at BNM-LNE.

KEY WORDS : ambient temperature, directional, emissivity, reflectance, spectral.

1. INTRODUCTION

For almost 20 years, BNM-LNE has constructed and improved instruments to measure infrared radiative properties of materials. These properties are used for pyrometry, thermography and heat balance calculation in many applications (research, industry processes, military applications). An infrared reflectometer was designed by BNM-LNE, some years ago, to measure the directional hemispherical spectral reflectance $\rho(\theta, hem, \lambda)$ of solid materials at ambient temperature. For opaque materials, the directional spectral emissivity $\varepsilon(\theta,\lambda)$ can be calculated from the measured reflectance using the relation : $\varepsilon(\theta,\lambda) = 1 - \rho(\theta, hem, \lambda)$.
The reflectometer which is described uses a novel optical arrangement (base on the Coblentz's arrangement) to collect the reflected radiation. Each source of uncertainty has been analysed and the overall uncertainty has been evaluated. The measurement results have been validated by comparison to other measurement techniques.

2. DESCRIPTION OF THE APPARATUS

Fig. 1 shows a general diagram of the apparatus. Two sources can be used for the generation of the incident beam, a lamp for short wavelengths $(\lambda < 3\ \mu m)$ and a black-body for longer wavelengths. A monochromator with 3 gratings or a set of interference filters are used to select the spectral band (between 0.8 µm and 14 µm). The radiation is modulated by a chopper before the monochromator or the filter. The incident beam is limited by a circular field stop and an aperture stop. The field stop determines the size of the spot on the specimen. A rotating flat mirror and a set of 5 flat stationary mirrors are used to focus the incident beam on the surface of the sample with one of the 5 possible angles of incidence (12°, 24°, 36°, 48°, 60°). The principle used to collect the reflected flux, whatever the spatial distribution of the reflected radiation, is based on the Coblentz's arrangement, but a set of 4 mirrors with spherical surfaces is used instead of a simple hemispherical mirror. The 4 mirrors were "cut" in an hemisphere as shown on Fig. 3. and Fig.4 ; Fig. 3. emphasizes the mirror with the entrance slots for the incident beam. Each of these 4 mirrors collects the radiation reflected in a quarter of the half space and focuses it at a point located in the same plane as the surface of the sample as illustrated on Fig. 5. The mirror M1 focuses the radiation on the point I1, the mirror M2 on the point I2 and so on. So the radiation reflected by the specimen is focussed by the 4 mirrors at 4 different points. The pyroelectric detector (PVDF detector with a sensitive surface 10 mm in diameter) is successively placed at the 4 focal points. So the sum of the 4 signals measured at the 4 focal points (the "sample" signal) is proportional to the total flux reflected by the specimen. When the detector is placed at one of the 4 focal points , 3 light traps are placed at the 3 other focal points to avoid interreflections. A mechanical rotating system carries the detector and the 3 light traps and place them successively at the 4 positions. A "reference" signal is measured with the incident ray focussed directly on the detector. The directional hemispherical reflectance is then given by the relation $\rho(\theta, hem, \lambda) = \frac{1}{\rho_{mirror}} \cdot \frac{S_{sample}}{S_{reference}}$ where S_{sample} is the "sample" signal, $S_{reference}$ is the "reference" signal, ρ_{mirror} is the spectral regular reflectance of the mirrors that collect the reflected radiation, λ is the wavelength and θ is the angle that defines the direction of incidence. This optical arrangement was chosen to limit the angle of incidence on the detector; that angle is lower than 38°. The reflectance of the 4 mirrors that collect the reflected flux has to be known to calculate the reflectance of the sample. Two plane mirrors where manufactured using the same process as for the spherical mirrors (same substrate, same treatment) and the specular reflectance of those mirrors was measured.

3. EVALUATION OF UNCERTAINTY

The method used to calculate the uncertainty is the one recommended by the CIPM in the Guide to the Expression of Uncertainty in Measurement [1]. Uncertainties of all the parameters used in the calculation of the reflectance have been quantified. The uncertainty sources are : the uncertainty on the reflectance factor of the collecting mirrors; the spatial non-uniformity of the sensitivity of the detector; the uncertainty on the linearity of the detection chain; the resolution of the detection chain; leakages of the reflected flux; the interreflection between the sample and the detector; the noise of the measured signals; the polarization of the incident radiation; the atmospherical absorption (difference of optical path between the "sample" measurement and the "reference" measurement); the differences between the regular reflectance of the mirrors that directs the incident beam on the sample ("incident" mirrors) and the "reference" mirror that directs the beam on the detector for the "reference" signal measurement.

The relative standard uncertainty on the reflectance factor of the collecting mirrors is 1% for wavelength between 0.8 µm and 1 µm and 0.5 % for wavelength above 1 µm.
The effect of the spatial non-uniformity of the sensitivity of the detector can't be calculated rigorously as the real spatial distribution of radiation on the detector is not known. The non-uniformity of the spatial sensitivity has been measured, the local sensitivity of the detector can change by about 10 % over the sensitive area. The ratio of a signal measured with the ray focused at the center of the sensitive area to a signal measured with almost all the sensitive area irradiated is equal to 1 +/- 0.025. So the standard relative uncertainty on the reflectance due to the non-uniformity of the spatial sensitivity is estimated to a level of 1.25 %. The linearity of the radiative measuring chain has been experimentally controlled using the flux addition method. The uncertainty due to linearity depends on the ratio "sample" signal to "reference" signal. The standard uncertainty due to linearity is 0.0025 for a ratio between 0.5 and 1; 0.005 for a ratio between 0.1 and 0.5; 0.01 for a ratio between 0 and 0.1. The uncertainty due to the resolution of the measurement is low compared to other sources of uncertainty, nevertheless that uncertainty is taken into account. The leakagess of the reflected radiation arise mainly from the 5 slots drilled in one of the collecting mirror. Light diffusion from the collecting mirrors, a tilt of the sample surface and a bad position of the sample surface can also generate leakages. For a perfectly diffusing sample the relative the leakages through the 5 slots is equal to 0.62 % of the reflected flux. For non-specular materials a correction of 0.31% is made to take into account those leakages of radiation and the related standard relative uncertainty on the reflectance is 0.16 %. The standard relative uncertainty evaluated for the leakages resulting from a tilt of the

sample surface, the diffusion on the collecting mirrors and a bad position of the sample is 0.2 %. So the overall standard relative uncertainty for the leakagess is 0.26 %. The intereflections between the sample and the detector produce the same relative increase of the "sample" signal and "reference" signal. Therefore, in theory, the effect of the intereflections on the measured reflectance is nul. The optical path for the "sample" signal measurement is 0.2 m longer than the one for the "reference" signal measurement (twice the collecting mirrors curvature radius). So the "sample" signal is affected by the atmospherical absorption in the spectral bands 2,5 µm to 3 µm and 5 µm to 7,5 µm due to the water vapor absorption and in the spectral band 4 µm to 4,6 µm due to the CO_2 absorption. The optical system is located in a closed chamber and a flow of dried air (dew point below – 20°C) is maintained during the measurements. The resulting standard relative uncertainty due to the atmospherical absorption is 0.25 % for the spectral bands 2,5 µm to 3 µm and 4 µm to 4,6 µm and is 0.5% for the spectral bands 5 µm to 7,5 µm. The 5 plane mirrors that direct the incident beam on the sample ("incidence" mirrors) are supposed to have the same reflectance as the plane mirror that directs the beam on the detector for the "reference" signal measurement ("reference" mirror). The set of the 6 mirrors were supplied from the same manufacturer so the substrate and the coating are the same. The differences of reflectance arise mainly from the difference of ageing between the mirrors. The angles of incidence are almost the same (within 6°) for the 6 mirrors. The standard relative uncertainty on the reflectance coming from a possible difference of reflectance between the "incidence" mirror and the "reference "mirror is evaluated to 0.5 %. In the same way, the angle of incidence on the plane rotating mirrors used to select the incidence is not the same for the "reference" signal measurement and for the "sample" signal measurement. The related standard relative uncertainty is 0.5 %. The noise on the measured radiative signals can be significant compared to the reference signal. So for each radiative signal, the measurements are repeated several times and the mean value and the standard deviation are calculated. The uncertainty on the "sample" signal to "reference" signal ratio is then calculated by combination of the standard deviations of the radiative signals. The polarization of the incident beam has been analyzed. When an interference filter is used for the spectral selection, the incident beam is almost unpolarized ; the relative difference between the 2 polarizations components is less than 2 %. When the monochromator is used, the beam is significantly polarized, the intensity ratio between the 2 polarization components can be larger than 2. Practically in most cases, the polarizer is not used because, when the incident beam is linearly polarized, the incident flux is almost divided by 2 and the signal to noise ratio is too low. So when the incident beam is not linearly polarized, the relevant standard relative uncertainty component is evaluated to 0 %, 1 %, 1.5 %, 2 % and 3.5 % respectively for the incident angles of incidence 12°, 24°, 36°, 48° and 60°. The uncertainty related to the spectral bandwidth used for the measurement is not taken in account. The spectral bandwidth is just given as a measurement parameter.

4. VALIDATION OF THE MEASUREMENTS

Measurements were performed on a white ceramic tile (SRM 2019C) supplied and certified by NBS (NIST) in 1983 for directional hemispherical reflectance in the spectral range from 250 nm to 2500 nm and at 6° incidence angle. The results from

NBS are given in ref. [2]. The results of the comparison of the measured reflectances are given in Tab. I. Measurements were also performed by BNM-LNE and by PTB [3] on the black paint Nextel Velvet Coating 811-21 in the spectral range from 4 µm to 14 µm ; the results are given in Tab. II. On the black paint, BNM-LNE measured the spectral directional hemispherical reflectance and then calculated the spectral directional emissivity while PTB measured directly the spectral normal emissivity by comparison of the spectral irradiance of the sample to that of a blackbody.

The differences of spectral reflectances between BNM-LNE and NIST on the high reflecting white ceramic tile are very low compared to the uncertainties. The differences between the spectral emissivities measured by BNM-LNE and PTB on the black paint are higher but remain in the uncertainty range.

Table I. Comparison of the results of BNM-LNE to those of NIST (NBS) on the white ceramic (SRM 2019c)

Wavelength (µm)	BNM - LNE Reflectance Incidence 12°	NIST (NBS) Reflectance Incidence : 6°	Difference LNE - NBS
0.8	0.855 +/- 0.032	0.854 +/- 0.01	0.001
1.0	0.844 +/- 0.027	0.851 +/- 0.01	-0.007
1.5	0.862 +/- 0.028	0.859 +/- 0.01	0.003
2.0	0.855 +/- 0.028	0.863 +/- 0.01	-0.008
2.5	0.826 +/- 0.028	0.838 +/- 0.01	-0.012

Table II. Comparison of the results of BNM-LNE to those of PTB on the black paint (Nextel Velvet Coating 811-21).

Wavelength (µm)	BNM - LNE Spectral emissivity Incidence 12°	PTB Spectral emissivity Incidence : 0°	Difference LNE - PTB
4	0.960 +/- 0.02	0.975 +/- 0.032	- 0.015
4.5	0.962 +/- 0.02	0.966 +/- 0.011	- 0.004
8	0.959 +/- 0.025	0.974 +/- 0.006	- 0.015
10	0.950 +/- 0.027	0.967	- 0.017
12	0.940 +/- 0.035	0.972	- 0.032
14	0.940 +/- 0.045	0.973	- 0.033

5. CONCLUSION

The uncertainties have been analyzed in detail. For low reflecting samples the main sources of uncertainty are the noise of the radiative signals. This can be explained by the low radiative detectivity of the pyroelectric detector and by the high sensitivity of this detector to mechanical and acoustical vibrations. For high reflecting materials, the main source of uncertainty is the non-uniformity of the sensitivity of the pyroelectric detector over the sensitive area.

The reflectometer developped by BNM-LNE allows to perform reliable directional spectral reflectance measurements on any solid material at ambient temperature with absolute uncertainties from 0.02 to 0.045 (level of confidence 95%). These levels of uncertainty are suitable for most of the industrial applications.

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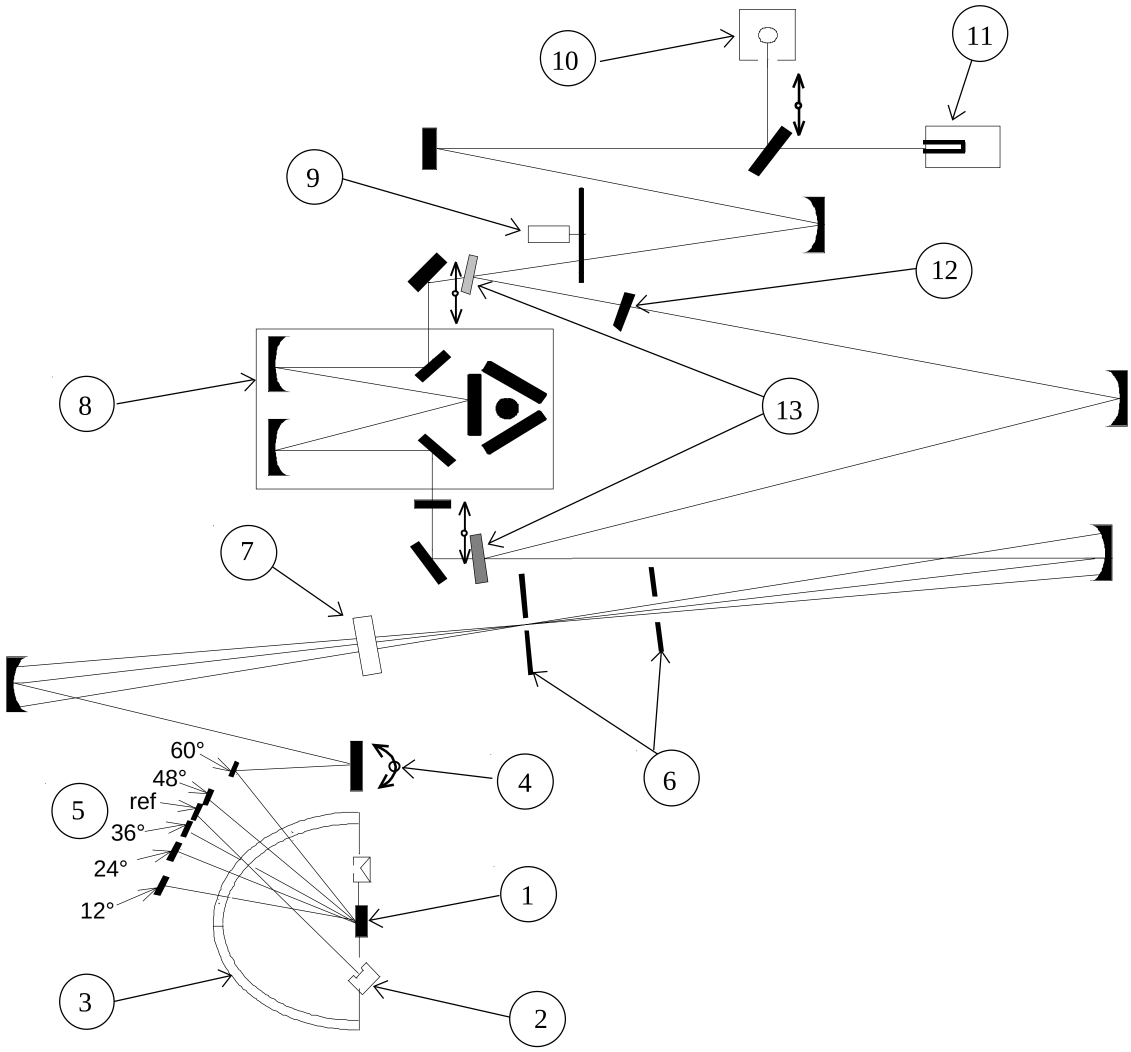

Fig.1. Diagram of the apparatus : (1) Sample; (2) Detector; (3) Set of 4 mirrors with spherical surface; (4) Rotating plane mirror; (5) Fixed plane mirrors for the selection of the angle of incidence; (6) Field stop and aperture limiting stop; (7) Polarizer; (8) Grating monochromator; (9) Mechanical chopper; (10) Lamp source; (11) Black-body source; (12) Interference filter; (13) Flat mobile mirrors for the selection of the monochromator or the interference filters.

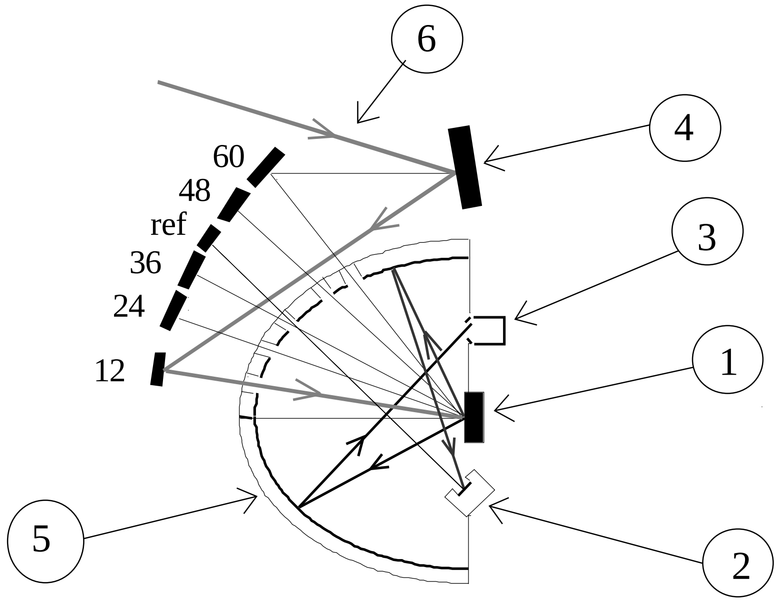

Fig.2. Collection of the reflected radiation : (1) Sample; (2) Detector; (3) Light trap; (4) Rotating plane mirror; (5) Set of 4 mirrors with spherical surface for the collection of the reflected flux; (6) Incident beam.

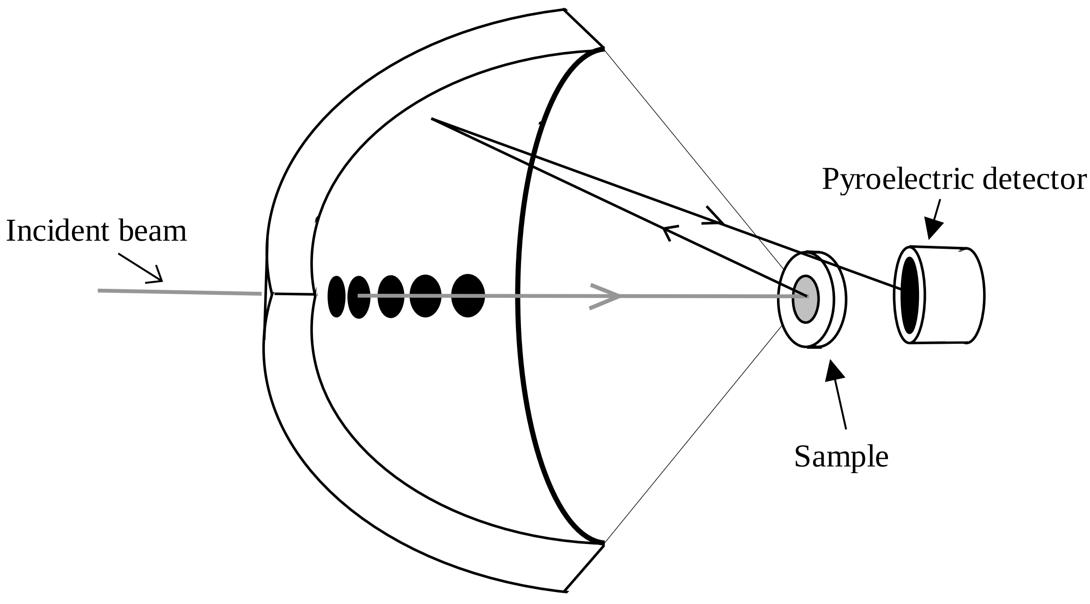

Fig.3. Focussing of the reflected flux on the detector

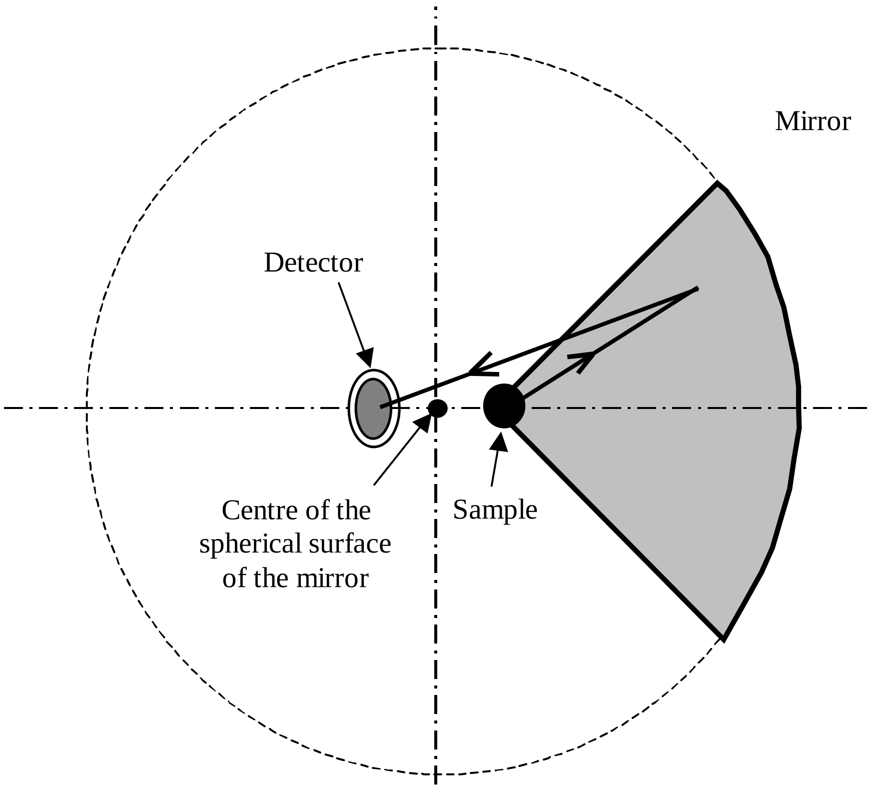

Fig.4. Focussing of the reflected flux on the detector

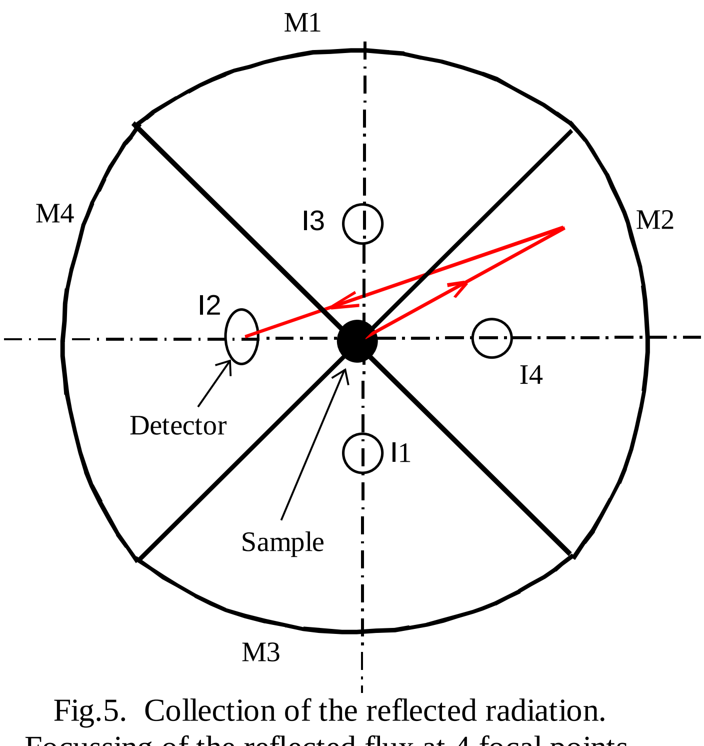

Fig.5. Collection of the reflected radiation.
Focussing of the reflected flux at 4 focal points.